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E. Bauer

Arizona State University

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EPITAXIAL GROWTH STUDIES BY LOW ENERGY ELECTRON MICROSCOPY

E. Bauer*

Department of Physics and Astronomy, Arizona State University, Tempe, AZ 85287; and
Physikalisches Institut, and Technische Universität Clausthal, D 38678 Clausthal-Zellerfeld, Germany

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Abstract

In spite of being a surface sensitive tool, low energy electron microscopy (LEEM) can also give information on interfaces. An example is the $\text{CoSi}_2/\text{Si}(111)$ interface. Most of the work discussed in this paper, however, makes use of the high surface sensitivity of LEEM which makes this method an ideal tool for the study of the early growth stages in epitaxy, in particular of the growth dynamics and of the influence of misfit on the growth mode. Two prototype substrates, Mo(110) and Si(111), and three representative deposit metals, Cu, Au and Co, are used to illustrate the large variety of phenomena which can occur in epitaxial growth.

Key Words: Low energy electron microscopy, epitaxy, phase transitions, surface dynamics.

Introduction

Can a surface science technique such as low energy electron microscopy (LEEM) give information on interfaces and layered structures? At first glance the answer seems to be no, because low energy electrons are believed to interact so strongly with matter that only the first few atomic layers contribute noticeably to the signal observed whether it is elastic [low energy electron diffraction (LEED)], inelastic [electron energy loss spectroscopy (EELS)], quasi-elastic [high resolution EELS (HREELS)], or due to secondary processes [Auger electron spectroscopy (AES)]. The basis of this belief is the so-called "universal curve" for the inelastic mean free path l_{ee} which has been derived from many experimental data and is also theoretically well supported. Significant deviations occur only for insulators and at very low energies below the plasmon excitation threshold E_{pt} . Below E_{pt} , electron-hole pair creation determines l_{ee} and the process depends strongly upon the band structure. In materials with a high density of unoccupied states, such as the transition metals with their narrow d electron bands, electron-hole pair creation has a high probability while in wide band gap materials this probability is low. Consequently, l_{ee} can vary strongly from one material to the next, for example with d band occupancy. Thus, in Au with its fully occupied d band, $l_{ee} \sim 2$ nm while in Gd, $l_{ee} \sim 0.25$ nm for electron energies within 5 eV from the vacuum level (Siegmann, 1992).

Obviously, the universal curve is not universal any longer below the plasmon threshold and the question whether or not electrons of a given energy can penetrate to an interface depends strongly on electronic structure and on E_{pt} . E_{pt} , which varies with plasmon energy, is usually in the energy range from 10 eV to 30 eV above vacuum level and is approximately the upper limit for large l_{ee} values. A good illustration for the long inelastic mean free paths at very low energies is the spin-polarized quantum size effect oscillations seen in Cu(111) layers on a Co(0001) layer up to at least 14 Cu monolayers corresponding to about 3 nm in thickness (Poppa *et al.*, 1993). It should be kept in mind, however, that such large penetration depths are possible only at

*Address for correspondence:

E. Bauer
Physikalisches Institut,
Technische Universität Clausthal,
D-38678 Clausthal-Zellerfeld,
Germany

Telephone number: (49) 5323-722144
FAX number: (49) 5323-723600

energies at which the crystal studied has no band gap in the incidence direction of the electron wave. Otherwise, the crystal acts as a reactive medium into which the electron wave can penetrate only by a distance of the order of the wavelength which is typically in the range from 0.2 to 0.4 nm at very low energies.

Summarizing this discussion, we can expect in many systems sufficiently large penetration depths in LEEM so that interfaces buried as deep as 2-3 nm below the surface can be imaged directly, provided we use energies below the plasmon threshold and stay away from band gaps. One condition has to be fulfilled, of course: the contrast of the interface features must be strong enough so that it is not overwhelmed by the contrast in the layer on top of it, whether it is diffraction contrast or quantum size effect contrast. This condition is fulfilled, for example, in indirect interface imaging via misfit dislocation contrast because the strain field of the dislocation penetrates far into the layer and changes locally the diffraction conditions in it and consequently the contrast. Also, interface steps may be visible in a similar manner provided their strain field extends far enough. Interfaces buried as deep as 70 nm have been imaged in this manner (Tromp *et al.*, 1993).

In general, however, LEEM is used to image the very surface and, in particular, the processes occurring on it which is possible because of the high intensity available at low electron energies. The principles of the technique, its instrumental aspects and its general applications have been discussed in several reviews (Bauer and Teliéps, 1987, 1988; Bauer, 1990, 1994a, 1994b; Veneklasen, 1992) and will not be described here again. Rather, we will concentrate on some studies of the early stages of epitaxial growth of metals on metals and semiconductors by discussing (i) three systems with different misfit, Cu, Au and Co on Mo(110) and W(110); (ii) three systems with different chemical interaction, Cu, Au and Co on Si(111); and (iii) some common growth modes.

Initial Growth and Stability of Cu, Au and Co on Mo(110) and W(110)

Provided that no alloying occurs, the six-fold symmetry of the face centered cubic (fcc) or the hexagonal close packed (hcp) (0001) plane has to be matched with the two-fold symmetry of the body centered cubic (bcc) (110) plane. Theory (van der Merwe, 1982) predicts, for all three systems, an initial pseudomorphic (ps) layer which transforms with increasing thickness into a misfitting layer with Nishiyama-Wassermann orientation (Cu, Co) or Kurdjumov-Sachs orientation (Au). At which thickness this occurs cannot be predicted by theory at present because the necessary atomic parameters are not

known. However the general growth mode (Volmer-Weber, Stranski-Krastanov or Frank-van der Merwe) follows, rather reliably, from elasticity and surface energy considerations (van der Merwe and Bauer, 1989). Also, the activated transition from metastable close-packed (cp) to ps islands observed in the submonolayer range of Co on Mo(110) is theoretically understood (van der Merwe *et al.*, 1994).

For a more detailed understanding, it is interesting to compare Cu and Co on the one hand with Au on the other. The atomic diameters in the metallic state of Cu (0.256 nm) and Co (0.250 nm) are much smaller than those of Mo (0.274 nm) and W (0.278 nm), that of Au (0.288 nm) is noticeably larger. Therefore, ps monolayer (ML) formation is not difficult in Cu and Co while Au can grow in a ps layer only by introduction of missing atomic rows. When equilibrated by deposition or annealing at high temperatures, these rows form a periodic array. The resulting LEED pattern is similar to that of a misfit dislocation array and has also been interpreted in this manner in the past. On W(110), the missing rows are parallel to the [001] direction, on Mo(110) parallel to the [112] and [112] directions although there is only a very small difference between the two materials in lattice constant and electronic structure. This illustrates how subtle differences can influence epitaxial growth. The influence of two equivalent missing row directions on the growth is clearly seen in the LEEM images of Au submonolayers quenched from the two-dimensional (2d) gas phase (Fig. 1) (Mundschau *et al.*, 1988).

As expected, Cu and Co grow in the submonolayer range on Mo(110) and W(110) in a very similar fashion: at low temperatures, via nucleation on steps and terraces; at high temperature, via step flow growth. The front is very sensitive to co-adsorption: in the absence of contamination, it is smooth; with contamination, it is fractal-like, similar to the theoretically predicted growth at low temperatures and seen in many STM studies. Upon completion of the ps ML, a second layer forms on Cu but the Co ML incorporates more Co until the cp ML is completed. The growth of the cp Co ML islands is highly anisotropic and limited to the terraces on which they nucleate, a phenomenon typical for many systems in which structural rearrangement of the existing layer must occur for further growth. The second and additional Co layers differ little in structure from the cp ML so that quasi-Frank-van der Merwe growth is possible up to about 450 K. At higher temperatures, however, when the equilibrium structure can be approached, Stranski-Krastanov growth occurs with large flat epitaxial Co islands which preferentially nucleate at substrate imperfections. Figure 2a illustrates this for a Co layer with an average thickness of about 10.3 ML deposited onto

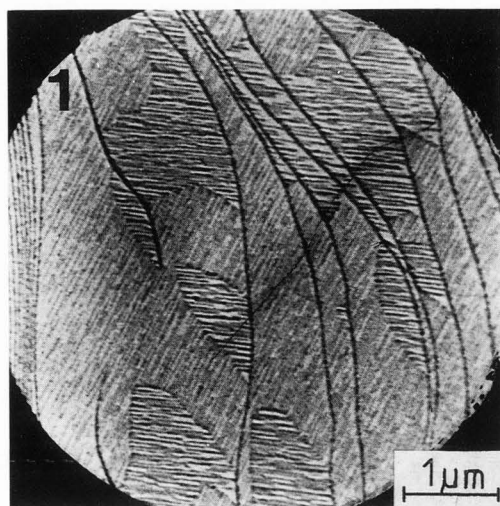


Figure 1 (above). Au submonolayer on Mo(110) quenched from the two-dimensional gas phase. The long lines are monatomic steps. Electron energy 14 eV.

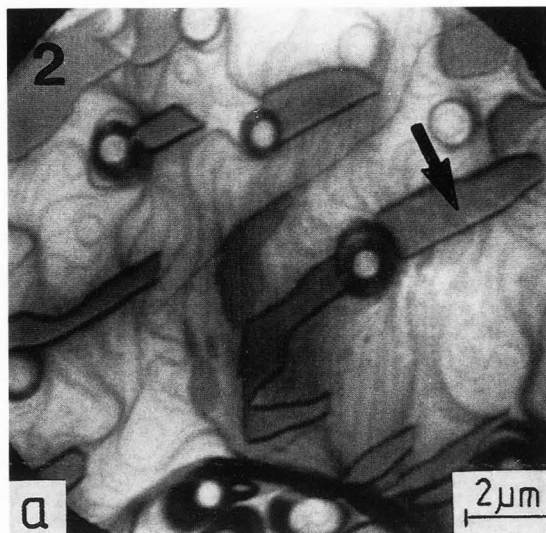
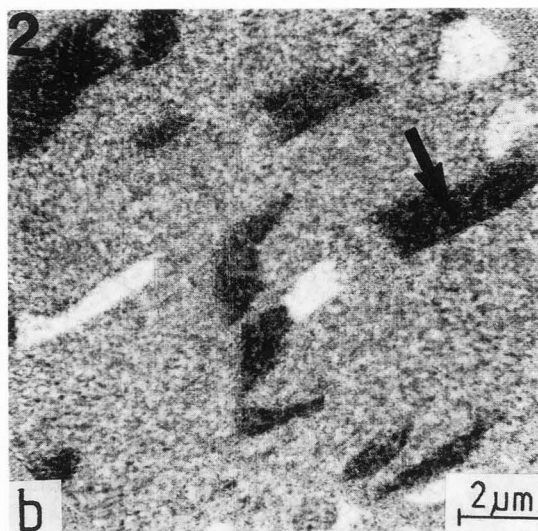


Figure 2 (at right, top). Three-dimensional Co islands on a W(110) surface covered with a Co monolayer grown by depositing about 10.3 monolayers of Co at 750 K: (a) is an ordinary LEEM image, (b) a spin-asymmetry image. One of the crystals is marked by an arrow. 2.0 eV.



W(110) at 750 K. The corresponding spin-polarized LEEM image in Figure 2b shows that these islands are all magnetized in one of two equivalent directions ("uniaxial anisotropy") (Pinkvos *et al.*, 1993). Deposition or annealing at still higher temperatures leads to dissolution of W or Mo in the three-dimensional (3d) islands. After thermal desorption of Co from the then round liquid alloy islands, the round W or Mo "mesas" seen in Figure 2 remain.

The growth of Cu on Mo(110) is more complicated due to the fact that the double layer may exist in three different structures: a metastable and a stable low temperature double layer and a high temperature double layer, the latter two reversibly converting into each other upon heating and cooling (Tikhov *et al.*, 1987). At low temperatures (< 600 K), the double layer consists of two cp Cu layers, and the growth kinetics is the same as in the ps → cp transition in Co MLs if the ps ML was completed before the second layer starts to grow. When the first ML still consists of many ps islands, the double layer islands evolve explosively before the ML islands have grown together. Growth of additional layers is always limited to the terraces on which nucleation

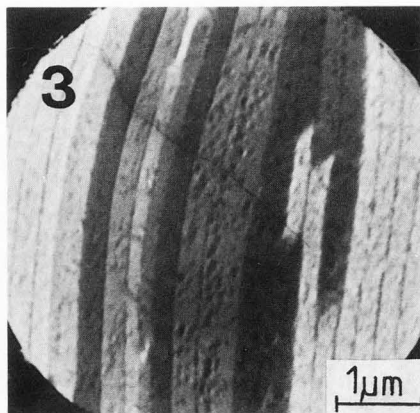


Figure 3. Cu multilayer on Mo(110) with different thickness on different terraces illustrating the quantum size effect. 4 eV.

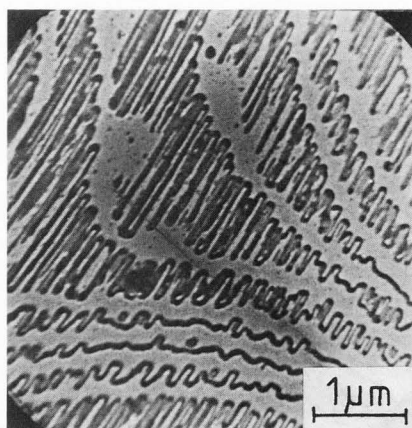


Figure 4. Cu double layer on Mo(110) at high temperatures with misfit strain relaxation structure. 3 eV.

occurred and spreads along these terraces. This can be followed in detail via the quantum size effect contrast (Fig. 3) (Mundschau *et al.*, 1989a). When a low temperature double layer is heated above about 600 K then some material is expelled and forms 3d crystals. Subsequent cooling leads to a double layer which consists of a ps first layer and a cp second layer with zero misfit in the Mo[110] direction. When heated again, a double layer, with zero misfit in the Mo[001] direction, forms above about 650 K. This "misfit-flip" transition between the two structures is completely reversible and occurs with significant hysteresis. Its kinetics can be followed nicely with LEEM. During high temperature growth, the initially formed second ML islands are strongly elongated along the zero misfit direction. Cu in excess of 2 ML forms large flat 3d crystals without distinct crystallographic shape.

Deposition or annealing at still higher temperatures ($T > 900$ K) causes dramatic changes in the surface structure: starting from the steps the surface breaks up into a hill and valley structure with long [001] steps (Fig. 4) (Mundschau *et al.*, 1988). The process is believed to be driven by stress relief in the one-dimensionally misfitting Cu double layer / Mo system. During Cu desorption, the surface reverts back to the smooth step structure of the clean surface. This example shows that at high temperatures the substrate may not be considered to be rigid any longer but participates strongly in misfit-driven structural rearrangements.

Chemical Interactions and Growth Modes: Au, Cu and Co on Si(111)

While in the previous section the influence of misfit was emphasized, the importance of chemical interactions

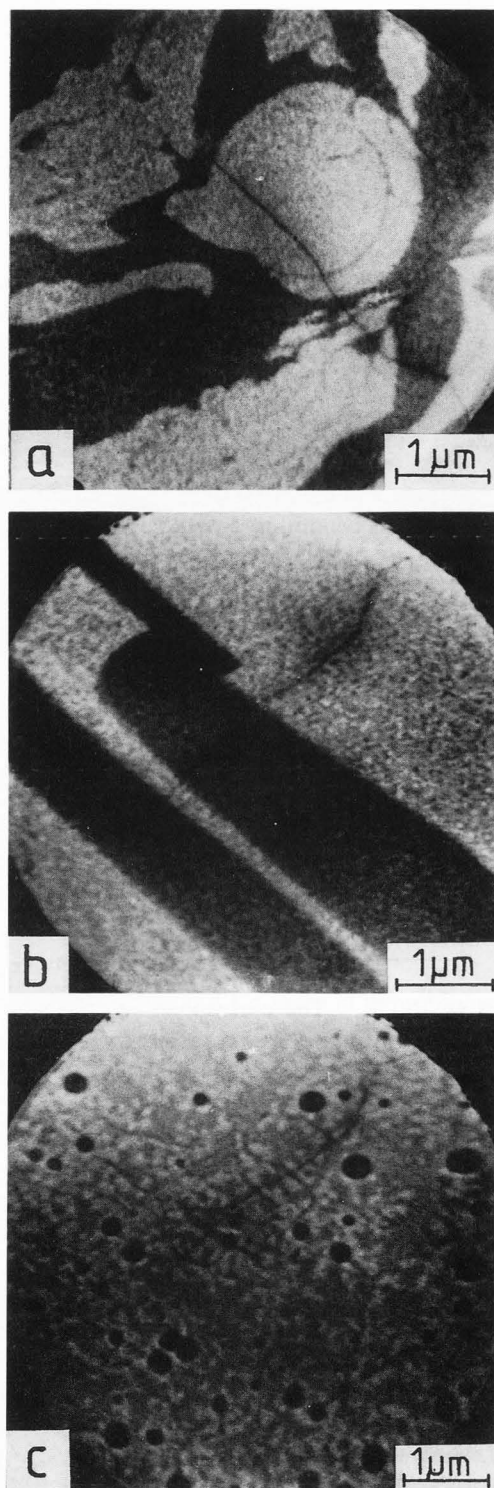


Figure 5. Au submonolayer structures on Si(111) obtained by cooling from high temperatures at fixed coverage: (a) (5×1) structure (bright regions) in (1×1) structure environment, 12 eV; (b) (5×1) structure (dark) in $(\sqrt{3} \times \sqrt{3}) R30^\circ$ structure environment, 3 eV; and (c) (6×6) structure (bright) plus three-dimensional Au particles, 1 eV.

will be illustrated now. Au forms a eutectic with Si and so does Cu but Cu also forms several stable silicides while in the case of Au, the stability of silicides is still not firmly established. Co also forms a number of silicides. Thus, the three systems seem to have some similarities but the question remains: to what extent do they form 2d silicides before 3d crystals appear. For Au and Cu, this question is settled by numerous studies of the 2d "silicides" (5×1) , $(\sqrt{3} \times \sqrt{3}) R30^\circ$ and (6×6) for Au and " (5×5) " for Cu, mainly by STM work. Nevertheless, LEEM is useful for the study of the growth kinetics either during deposition or upon cooling from the disordered state.

Deposition studies of Au on Si(111) at elevated temperatures (Swiech *et al.*, 1991) clearly show significant differences in the growth kinetics of the (5×1) and of the $\sqrt{3}$ structures, the former starting at steps and at (7×7) domain boundaries and growing with well-defined shapes, the latter without concern for steps, in large islands and in a very irregular manner. The (6×6) structure finally starts again preferentially along steps but does not appear before a large number of 3d Au particles have formed.

Growth of these structures at fixed coverage during cooling from the high temperature phase is quite different. When the (5×1) structure coexists with the (1×1) or the (7×7) structure, it spreads with large velocity across the terraces on which it nucleated when the temperature is lowered. If the undercooling is small, the growth suddenly stops. In order to continue growth, additional undercooling is necessary which varies from terrace to terrace. The step and terrace structure in this process is still similar to that of the substrate before deposition and does not change during cycling through the transition (Fig. 5a). If the (5×1) structure coexists with the $\sqrt{3}$ structure, the steps are strongly rearranged to form wide terraces on which the structure grows in a manner very similar to that described before for the growth of the cp Co ML on W(110) and of Cu multilayers on Mo(110). Again, the growth suddenly stops if the undercooling is small and continues step-wise with further undercooling steps (Fig. 5b). Repeated cycling through the transition does not cause significant changes in the domain and step structure. This is not true for the (6×6) structure. When this structure is formed by slow deposition at about 600 K, the nucleation rate is low and relatively large islands grow. Cycling through the $(6 \times 6) \leftrightarrow \sqrt{3}$ transition causes a strong increase in the island density and decrease in the perfection of this structure (Fig. 5c). Simultaneously, the microstructure of the Si surface is changed significantly, in part due to movements of the coexisting Au crystallites / Au-Si eutectic droplets.

At higher coverages and temperatures, these 3d par-

ticles can move over large distances across the surface, reacting with the substrate and leaving reaction trails behind. At fixed coverage, the number of particles decreases and their size increases with time as a consequence of this mobility, in part also due to Oswald ripening. Temperature changes cause significant changes of the particle shapes due to temperature-dependent wetting, unwetting and phase-separation of the Au-Si eutectic. Because of all these processes, it is difficult to grow continuous Au films on Si at high temperature.

A similar statement can be made for Cu on Si(111). In this system only one 2d silicide, the " (5×5) " structure which actually is incommensurate [(5.3×5.3) to (5.6×5.6) has been reported] grows initially at high temperatures. The formation kinetics of this structure is complicated. Nucleation at steps and terraces has been observed, and growth may occur either along or across steps. After the completion of the " (5×5) " structure, 3d Cu silicide crystals nucleate in various shapes and the substrate steps agglomerate into faceted multiple steps. An example is shown in Figure 6 (Mundschau *et al.*, 1989b). At very high temperatures, above the Cu-Si eutectic temperature, the smallest Cu silicide crystals become mobile and produce reaction trails similar to the Au-Si eutectic droplets. Thus, in many respects, Cu on Si is similar to Au on Si, differing mainly in the solubility, in the number of 2d structures, and in the stability and composition of the 3d particles. In both cases, a 2d layer is formed before 3d particles nucleate.

For Co on Si(111), the situation is quite different. It has been known for some time that continuous well-ordered CoSi_2 films can be grown at elevated temperatures only if a thin Co silicide "template" layer was first deposited at low temperature. Otherwise, films with many pinholes or 3d crystals form. This tendency of CoSi_2 not to wet the substrate has been attributed to a too large interfacial energy in spite of the small misfit which leads to such a large spacing of the misfit dislocations that they can be resolved by LEEM. Figure 7 (Bauer *et al.*, 1989) shows a LEEM image of a CoSi_2 layer consisting of large flat 3d crystals in which the dislocations are visible via strain contrast in spite of the high electron energy. Another example of interface imaging is that of Ag islands on Si(100) (Tromp *et al.*, 1993). The question, whether or not there is a 2d silicide between the 3d crystals, can be checked best by annealing at very high temperatures at which the equilibrium configuration can be approached. The result of such an experiment is shown in Figure 8 (Bauer *et al.*, 1991). A CoSi_2 layer was heated to such high temperatures that it transformed into individual CoSi_2 crystals and a significant amount of Si sublimed. CoSi_2 sublimes slower than Si so that large hillocks are formed, topped by CoSi_2 crystals. The surrounding surface has mostly

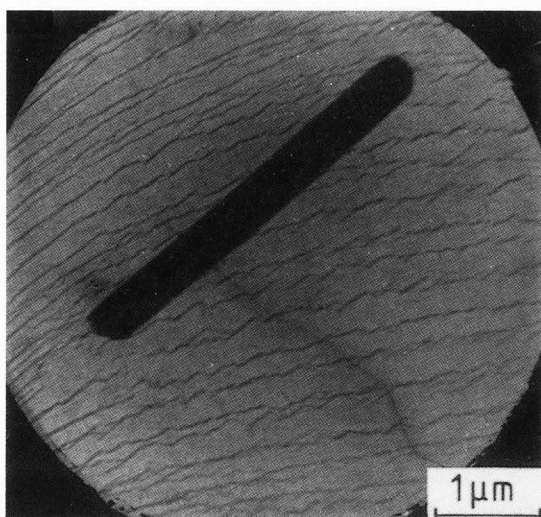


Figure 6. Cu layer on Si(111) grown at high temperature showing faceted steps and a three-dimensional Cu_3Si crystals. 4 eV.

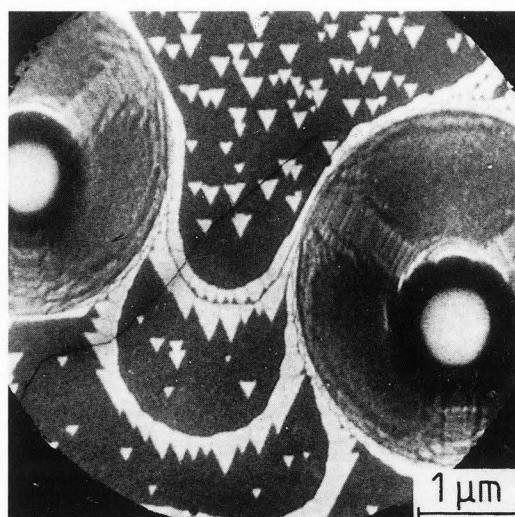


Figure 8. CoSi_2 crystals on top of hillocks on a Si(111) surface heated to Si sublimation temperatures. 10 eV.

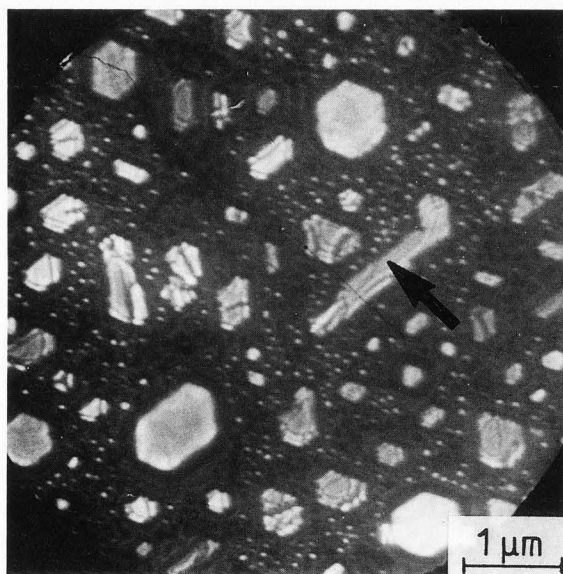


Figure 7. CoSi_2 layer on Si(111) annealed at high temperature consisting of flat three-dimensional crystals with misfit dislocations. Some well-separated dislocations are indicated by an arrow. 30 eV.

(1 × 1) structure with (7 × 7) crystallites only along the steps and locally on the terraces due to rapid quenching. Upon slow cooling, however, the free surface can be converted completely into the (7 × 7) structure. Thus, there is no 2d silicide layer and CoSi_2 grows in the Volmer-Weber growth mode. This is not due to the interfacial energy but a consequence of the large surface energy of the refractory CoSi_2 .

Discussion

The examples discussed above show much of what had already been known before the LEEM studies but contain also a considerable amount of new information, in particular, about the growth kinetics. Only three of these new findings will be discussed briefly here: (i) the impurity-blocked step flow growth; (ii) the anisotropic step flow growth; and (iii) the structural-rearrangement-hindered growth. Impurity-blocked step flow growth can be seen in Cu and Co layers on Mo(110) and W(110) surfaces. When steps and terraces are clean and no co-adsorption occurs during deposition, smooth step flow growth is observed. The growth velocity is determined by the supply of atoms which depends upon terrace width and step curvature. Both, concave and convex step regions may grow preferentially, the one, in order to reduce the step length, the other, because of the increased supply. For smooth step flow to occur, sufficient mobility must not only exist normal to the propagating step but also parallel to it, and the atoms must attach to the energetically most favorable sites. This is not possible when the growth is highly anisotropic as in the case of Au on Mo(110). In this case, the attachment probability depends upon the orientation of the growing step and only the tendency to minimize the total free step energy keeps the step from developing long needles.

Rough step growth also occurs when the original substrate or the growing steps are partially blocked by impurities such as C, O or CO which cannot easily be displaced by the arriving metal atoms. Localized decoration of the substrate steps is then seen along with

fractal-like growth during the spreading of the layer across the terrace. The transition from smooth to fractal-like growth obviously depends upon the relative arrival rate of metal and residual gas molecules and upon their bonding strength not only to the propagating step but also to the substrate. CO, for example, is weakly adsorbed on Cu but strongly on Mo and W. During growth on Mo and W, Cu displaces CO so that the CO density increases not only due to supply from the residual gas but also by compression of the CO already adsorbed. Layer growth, thus, proceeds on an increasingly contaminated surface. Only if the substrate temperature is high enough so that no growth-blocking impurities can be adsorbed is smooth growth possible. On W and Mo, the temperatures at which C, O and CO are not adsorbed are too high for metal condensation. Thus, smooth growth is possible only under very clean conditions. On other, more weakly adsorbing surfaces such as Cu, Ag or Au surfaces, this temperature is easily accessible. Not all fractal-like growth at low temperatures reported in the literature is, therefore, a proof of the theoretically predicted fractal growth.

Structural rearrangement-hindered growth is, at first glance, a very startling phenomenon. It can be seen best during the growth of a new structural phase across the surface either with increasing coverage or changing temperature, both in the submonolayer and in the multilayer region. Au/Si(111) and Co/W(110) are examples of the former, Cu/Mo(110) of the latter situation. The same type of growth can also be seen in island layers during structural transitions, for example, at the beginning of the second layer of Cu on Mo(110) at low temperatures. Qualitatively, this growth mode can be understood easily. The formation of a new structural phase usually requires nucleation. In order to overcome the nucleation barrier, a certain supersaturation is necessary. Once the new phase has nucleated on a given terrace, it can spread rapidly across the terrace scooping up the atoms within their diffusion distance, also across the steps. Further growth proceeds either at a rate determined by the deposition and diffusion rate or after some interruption until the necessary supersaturation has been reached to overcome some kind of growth barrier, for example, for restructuring the existing layer (first Cu layer on Mo(110) or ps Co layer on W(110)) or for displacement of blocking adsorbates. It should be noted that in most cases studied, growth was limited to the terraces on which nucleation occurred and that the growth rate on these terraces was so high that rapid diffusion across the steps must have occurred. A consequence of this growth mode is that, in spite of local monolayer-by-monolayer growth, the average surface may become very rough if the nucleation rate varies strongly from terrace to terrace.

Summary

This review tried to give an idea what LEEM can contribute to the understanding of interface formation and the dynamics in layered structures. Particular attention was paid to heteroepitaxial growth modes which can differ significantly from homoepitaxy because of the stored strain energy in the layer. The possibilities of studying buried interfaces with LEEM have hardly been explored and used up to now. Spin-polarized LEEM (SPLEEM), which is still in its infancy, may open up a new way to study interfaces involving magnetic materials. The qualitative nature of the results reported here is not due to limitations inherent to LEEM but a consequence of the only qualitative control of the experimental conditions, mainly temperature, deposition rate, and residual gas pressure, and the large amount of work needed to extract quantitative information from videos. Both problems will certainly be overcome in future work.

Acknowledgments

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Editor's Note: All of the reviewer's concerns were appropriately addressed by text changes, hence there is no **Discussion with Reviewers**.